

# Valorisation of Industrial Waste for the Development of Fire Resistant Material to Be Used as Filler in Paints and Varnishes

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# Abstract

The development of a Feslag based geopolymer [1] and its use is further examined. The developed geopolymer is used as a filler in paints, varnishes and plasters. The physical and thermal properties of those products are measured. It is shown that the materials present adequate mechanical strength, physical and thermal properties. All developed materials were subjected to thermal loading with the modification of a standardized passive fire protection test. The plaster succeeded to the test without failing in any of the criteria concerning the temperature in the unexposed surface of the specimen and its internal integrity. Further plaster testing on elasticity, open time, gloss value, alkali resistance and  $CO_2$  carbonation protection is also performed on developed plasters with very positive results.

## **Subject Areas**

Material Experiment

## **Keywords**

Geopolymer, ETICS Plaster, fire Resistant Filler, Carbonation Protection

# **1. Introduction**

The term "paints" defines materials or mixtures of materials that are in a liquid to very thick form (with a fluidity of paste) and are applied to a surface with a brush, roller or by the spraying method [2]. After their application to the surface, they dry and harden under the influence of physical, chemical or physico-chemical agents, providing a coherent coloured or not and well adhered to the surface opaque film, which confers decorative, protective and/or special technical properties. The term "coating" refers to the product that is based on organic, synthetic materials, and may contain coating colours (pigments), additives, fillers and organic solvents. After its deposition, it forms a film, which is able to protect and decorate the surface. Essentially, coating is the broadest concept of colour and refers to any product intended for the protection and decoration of surfaces. Finally, as "varnish" is considered the coating that does not include coating colours (pigments) and fillers. It could be considered as "clear paint" and be included in the colours within the more general concept of coatings.

The main components of any coating system are pigments, fillers, (solvents, mainly water), additives and the base (emulsion). Fillers, in addition to reducing costs, may also improve some properties, such as anti-corrosion, adhesion and film strengths. In previous study [1] we developed a filler based on Fe slag geopolymer. Fire tests acc. to ISO 834 to concrete specimens protected by 3cm thick Fe slag geopolymer were successful. (Figure 1)

In this study we test additional fire resistant [3] [4] [5] and concrete protection [6]-[15] from CO<sub>2</sub> properties of coatings containing geopolymer filler based on potassium Fe slag. We developed a plaster (ETPS) with Vinyl Acetate/Ethylene water dispersion as base emulsion and the new geopolymer filler, keeping all other materials same (to the product already commercializing). Additionally, we developed same plaster with metakaolin geopolymer filler as a benchmark (ETPMK). Small scale fire tests according to ISO 834 on samples  $40 \times 40 \times 20$ cm (20 cm concrete + 3 mm plaster) for the commercial product already in the market did not pass the criteria (temperature exceeded 180°C).



Figure 1. Fire protection test of the potassium based geopolymer used as a filler.

## 2. Experimental

## 2.1. Materials

Results on evaluation of the developed plasters characteristics is presented in **Table 1** and **Table 2** (0 Worst to 5 Best). **Figure 2** shows the plasters containing geopolymer filler.

Elasticity measurement was made with a Conical Mandrel Test device according to ISO 6860. For this specific measurement, the materials are applied to metal surfaces and with the use of the instrument they are rotated under pressure on a metal cone. The finished surface is evaluated based on standards from 0 (Worst) to 5 (Best). (Figure 3)

Resistance to alkalis (cleanability), was performed according the Greek ELOT 788 (DIN EN 53788). Flat glass tiles, 10 cm  $\times$  20 cm and 5 mm thick, were coated with the plasters and left to dry for 120 h. Instead of depositing drops on the surface, strength test was done by continuous immersion. The immersion was done in an aqueous solution of NaOH 0.5% for 4 hours. This was followed by rinsing with water and drying for 2 h. The result of the measurement is Pass or Fail. The specimen fails the test if a change in the shade of the coating occurs. (Figure 4)

Table 1. Plasters evaluation.

Characteristics	ЕТРМК	ETPS
Pigment binding power	5	4
Open time	4	5
Minimum Film Forming Temperature (°C)	4	3
Adhesion	5	5
Stability	5	5
Elasticity	4	5
Colour retention	3	4.5
Cost	4	3
TOTAL	34	34.5

Table 2. Plaster physical characteristics.

ЕТРМК	ETPS
VAE	VAE
14%	14%
MK geopolymer	Feslag geopolymer
1.74	1.77
28%	28%
82.09	81.50
	<b>ETPMK</b> VAE 14% MK geopolymer 1.74 28% 82.09



**Figure 2.** Plasters containing geopolymer filler (a) left with metakaolin filler, and (b) right with Feslag geopolymer filler.



Figure 3. Elasticity measurements on developed plasters.



Figure 4. Alkali resistance test.

Determination of gloss value of the coatings was measured with A Micro-TRI-Gloss Glossmeter according to DIN 67530. To measure the gloss, the materials were applied on concrete cubic specimens and 10 measurements were taken at different points on the surface, for each incidence angle of 20°, 60° and 80°. The final result is obtained as the average of the measurements. (**Figure 5**)



Figure 5. Gloss value measurement.

#### 2.2. Analysis and Tests

Finally, fire resistance tests according to ISO 834 and carbonation protection tests according to EN 1504-2 were performed on concrete samples for the two plasters containing geopolymer filler as a fire resistant additive.

#### 3. Results and Discussion

# **3.1 Fire Tests**

#### 3.1.1. ETPMK Plaster

ETPMK plaster was applied at 3 mm thickness on a  $40 \times 40 \times 20$  cm concrete slab. 28 days after curing the fire test was performed. Compressive strength tests of 50 mm diameter cores of the concrete slab, showed that the residual compressive strength is 56.2 MPa from 57.1 MPa (28 days) which was in the original—before the test—slab concrete.

The ETPMK plaster succeeded in all test criteria (no spalling, interface temperature lower than 180°C, concrete slab integrity and mechanical strength retained after fire exposure). (**Figure 6** and **Figure 7**)

Max temperature in the plaster/concrete interface reached 159°C after two hours.

#### 3.1.2. ETPS Plaster

ETPS plaster was also applied at 3 mm thickness on a  $40 \times 40 \times 20$  cm concrete slab. 28 days after curing the fire test was performed. Compressive strength tests of 50 mm diameter cores of the concrete slab, showed that the residual compressive strength is 56.6 MPa from 57.4 MPa (28 days) which was in the original—before the test—slab concrete. (Figure 8 and Figure 9)

Max temperature in the plaster/concrete interface reached 174°C after two hours.

#### 3.1.3. Carbonation Tests According to EN 1504-2

Because the carbonation of concrete under ambient conditions occurs at very slow rates, due to the low concentration of carbon dioxide in the atmosphere, accelerated carbonation measurements are carried out in a laboratory in special chambers. The concentration of carbon dioxide inside such a chamber is much higher than that of the atmosphere and thus we can have results in a shorter period of time regarding the resistance of a concrete to the phenomenon of carbonation.



Figure 6. Time temperature chart for the ETPMK plaster.



Figure 7. ETPMK plaster (a) before, (b) during, (c) after and (d) concrete slab after test.



Figure 8. Time temperature chart for the ETPS plaster.



Figure 9. ETPS plaster (a) before, (b) after and (c) concrete slab after test.

Procedure based on the EN 1504 standard is as follows:

Three concrete cubes are cast and hardened for 28 days according to EN 12390-2. They are then covered with the corresponding coating and the samples are left in a laboratory air environment for 14 days and then placed in the carbonization chamber. The concentration of carbon dioxide inside the storage chamber is at a percentage by volume of  $3.00 \pm 0.10\%$ , the temperature (20 ±

2)°C and the relative humidity  $(57 \pm 3)$ % for periods up to 70 days. After 7, 28 and 70 days of storage in the chamber, at the end of each exposure period, one of the cubes is split in half (or a 50 mm slice is cut from each prism) and the char depths are measured. On each prism slice or half of each cube, eight carburization depths are measured and averaged over the four sides. Using measurements taken at fixed hours, the carbonation rate is expressed in mm/days. For the measurement, a phenolphthalein indicator solution is used (1 g of indicator in 70 ml of ethanol and 30 ml of distilled water), which is sprayed on the concrete surfaces resulting from the cut.

According to procedure above, we tested ETPMK, ETPS plasters with geopolymer fillers based on metakaolin and Feslag respectively, ETPT plaster with chalk as filler (reference commercial plaster) and ETPPF plaster with aluminium hydroxide as fire resistant filler (already used in paints as a reference also). *ETPT and ETPPF plasters failed the ISO* 834 *fire tests at* 3 *mm thickness.* (Figure 10 and Figure 11)

 $CO_2$  exposure results are presented in following **Table 3**.



(a)



Figure 10. Plaster covered concrete cubes (a) before and (b) after 28 days exposure.



Figure 11. Carbonation depth over time.

Table 3.	CO,	penetration	depth	over	time.

Plaster	7 days carbonation depth (mm)	28 days carbonation depth (mm)	70 days carbonation depth (mm)
Uncovered concrete (reference)	1.54	6.16	11.80
ЕТРМК	0.00	0.00	1.90
ETPS	1.07	4.29	8.50
ETPPF	0.48	1.92	3.80
ETPT	0.62	2.46	4.90

# 4. Conclusions

Fire resistant plasters were developed using as filler, geopolymers based on metakaolin or Feslag. Properties such as elasticity, resistance to alkalis (cleanability), gloss value, resistance to fire and carbonation protection to concrete were studied for the developed plasters but also for reference products already available in the market.

- Results of all tests performed are presented in Table 4.
- ETPS plaster contains the Feslag geopolymer filler.
- ETPMK plaster contains the metakaolin geopolymer filler.
- ETPT plaster contains talk as filler.
- ETPPF plaster contains aluminium hydroxide as filler.

The Feslag filler geopolymer plaster developed offers a value for money alternative to commercially available fillers, offering at same time fire resistance to building constructions and some protection of  $CO_2$  attack on concrete surfaces. The low cost-low investment technology of geopolymers, is again proved to have further development opportunities and great field of applications on applied building materials chemistry.

#### Table 4. Properties of all plasters tested.

	TOP PLASTER ETPS	TOP PLASTER ETPMK	TOP PLASTER ETP-T	TOP PLASTER ETPPF
(Fire resistant) filler	Feslag geopolymer	metakaolin geopolymer	Talc. Reference plaster already available, no filler	Aluminium hydroxide
%w/w filler	28%	28%	0%	28%
Total P.V.C %	81.50	82.09	81.30	81.40
Emulsion	VAE	VAE	VAE	VAE
%w/w emulsion (50% solids)	14%	14%	14%	14%
Viscosity (cP)	17,000 - 18,000	17,000 - 18,000	17,000 - 18,000	17,000 - 18,000
Density (gr/ml)	1.770	1.740	1.760	1.750
Open time 0 - 5 (5 best/0 worst)	5	5	5	4
Elasticity 0 - 5 (5 best/0 worst)	4.5	4.5	5	5
Gloss (20°/60°/80°)	0.3/0.7/0.1	1.2/1.9/0.3	1.1/1.7/0.2	1.1/1.7/0.2
Alkali resistance	PASS	PASS	PASS	PASS
ISO 834 fire test	PASS	PASS	FAIL	FAIL
Carbonation protection (70 days CO <sub>2</sub> penetration depth (mm))	8.50	1.90	4.90	3.80

## **Next Steps**

The developed material (ETPS) will be further tested in large scale test according to EFNARC for its fire resistance properties according to RWS fire curve, for tunnel applications.

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# **Conflicts of Interest**

The authors declare no conflicts of interest.

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